

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR)

**10/031998**

INTERNATIONAL APPLICATION NO.

**PCT/US00/16234**

INTERNATIONAL FILING DATE

**13 JUNE 2000 (13.06.00)**

PRIORITY DATE CLAIMED

**17 JUNE 1999 (17.06.99)**

TITLE OF INVENTION

**PRESERVATION OF PAPER AND TEXTILE MATERIALS**

APPLICANT(S) FOR DO/EO/US

**BIRCHENALL, Andrew Kelsey**

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to being national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b)) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19<sup>th</sup> month from the earliest claimed priority date.
5. ☒ A copy of the International Application was filed (35 U.S.C. 371 (c) (2))
- a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau.
- b. ☐ has been transmitted by the International Bureau.
- c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☐ A translation of the International Application into English (35 U.S.C. 371 (c) (2)).
7. ☒ A copy of the International Search Report (PCT/ISA/210).
8. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c) (3))
- a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
- b. ☐ have been transmitted by the International Bureau.
- c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
- d. ☒ have not been made and will not be made.
9. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
10. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
11. ☒ A copy of the International Preliminary Examination Report (PCT/IPEA/409)
12. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

**Items 13 to 18 below concern document(s) or information included :**

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☐ A **FIRST** preliminary amendment.  
A **SECOND** or **SUBSEQUENT** preliminary amendment.
16. ☐ A substitute specification.
17. ☒ A change of power of attorney and/or address letter.
18. ☒ Certificate of Mailing by Express Mail.
19. ☐ Other items or information:

**17. General Power of Attorney****18. Express Mailing Label No.: EJ376014970US**

20. The following fees are submitted

**BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :**

- ☒ Search Report has been prepared by the EPO or JPO \$890.00
- ☐ International preliminary examination fee paid to USPTO (37 CFR 1.482) \$710.00
- ☐ No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) \$740.00
- ☐ Neither international preliminary examination fee paid to USPTO (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$1,040.00
- ☐ International preliminary examination fee paid to USPTO (37 CFR 1.482) And all claims satisfied provisions of PCT Article 33(2)-(4) \$ 100.00

CALCULATIONS PTO USE ONLY

**ENTER APPROPRIATE BASIC FEE AMOUNT =****\$890.00**Surcharge of **\$130.00** for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492 (e)). ☐ 20 ☐ 30**\$0.00**

| CLAIMS                                          | NUMBER FILED | NUMBER EXTRA | RATE                     |               |  |
|-------------------------------------------------|--------------|--------------|--------------------------|---------------|--|
| Total Claims                                    | 9 - 20 =     | 0 x          | \$18.00                  | <b>\$0.00</b> |  |
| Independent Claims                              | 3 - 3 =      | 0 x          | \$80.00                  | <b>\$0.00</b> |  |
| Multiple Dependent Claims (check if applicable) |              |              | <input type="checkbox"/> | <b>\$0.00</b> |  |

**TOTAL OF ABOVE CALCULATIONS =****\$0.00**Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) (check if applicable). ☐**\$0.00****SUBTOTAL =****\$0.00**Processing Fee of **\$130.00** for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492 (f)). ☐ 20 ☐ 30**\$0.00****TOTAL NATIONAL FEE =****\$890.00**Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable). ☐**\$0.00****TOTAL FEES ENCLOSED =****\$890.00**Amount to be :  
refunded \$

Charged \$

- ☐ A check in the amount of \_\_\_\_\_ to cover the above fees enclosed.
- ☒ Please charge my Deposit Account No. **04-1928** in the amount of **\$890.00** to cover the above fees.
- ☒ The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment to Deposit Account No. **04-1928** a duplicate copy of this sheet is enclosed.

**NOTE : Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (CFR 1.37(a) or (b)) must be filed and granted to restore the application to pending status.****SEND ALL CORRESPONDENCE TO:**

**SIEGELL, Barbara C.**  
**E. I. DU PONT DE NEMOURS AND COMPANY**  
Legal Patent Records Center  
1007 Market Street  
Wilmington, Delaware 19898  
United States of America

SIGNATURE

**SIEGELL, BARBARA C.**

NAME

**34,015**

REGISTRATION NUMBER

DATE

**Nov. 7, 2001**

10/031 998

TITLE

## PRESERVATION OF PAPER AND TEXTILE MATERIALS

FIELD OF THE INVENTION

The present invention relates to the preservation of paper articles (e.g., books, manuscripts, documents) and textiles articles (e.g., paintings on canvas, clothing, etc.) through the application of an amorphous fluoropolymer by, for example, spraying, dipping or brushing the article to be preserved with a solution of the fluoropolymer.

BACKGROUND

Commonly owned and copending PCT International Application No. PCT/US98/26903 discloses coating a substrate (e.g., a metal, ceramic or composite) including the application of a fluoropolymer solution to seal pores.

The use of fluoropolymer dispersions to coat and protect paper and fabrics is known (see e.g., U.S. Patent Nos. 4,742,140 and 5,674,961). Generally, these dispersions are comprised of particles in the neighborhood of 80 to 400 nm in diameter in an aqueous medium. The particles are not intended to fully or uniformly coat the fibers of paper or fabric. Also, because they are generally aqueous dispersions, items containing water-soluble dyes would be damaged by contact with water.

WO A 92/10532 teaches fluorinating the surface of polymers by deposition of fluorocarbons from solution. WO A 97/19224 is a process for preserving paper by polymerizing polycondensates *in situ*. GB A 007 981 discloses lamination of paper or textile articles. US 5,509,736 describes impregnation of paper with particles of oxides which hydrate to bases.

SUMMARY OF THE INVENTION

The present invention provides a method for strengthening a cellulosic paper or textile article, comprising the steps of (a) applying to the article a solution of an amorphous fluoropolymer in a perfluoroalkane solvent; and (b) drying the article so that the solvent is essentially removed.

The present invention also relates to a strengthened cellulosic paper or textile article comprising (i) a fibrous cellulosic paper or textile substrate and (ii) amorphous fluoropolymer interconnecting fibers of said substrate.

BRIEF DESCRIPTION OF THE DRAWING

Figure 1 represents a plot of data from Table 1.

DETAILED DESCRIPTION

The present invention employs amorphous fluoropolymers which, for application, are dissolved in a perfluoroalkane solvent. Articles to be preserved are treated with the fluoropolymer solution by any suitable method, including but

not limited to dipping, spraying and brushing. The article may be, but is not limited to, a book, manuscript, paper, fabric, article of clothing, painting, and the like. Normally, the amorphous fluoropolymer is used substantially transparent, and consequently there is no substantial difference in appearance between the treated article and the untreated article.

When fluoropolymer is deposited from solution onto a non-porous surface, a coating of about 5 to 20  $\mu\text{m}$  thick typically results. This thickness is related to the concentration of the solution used in the deposition. Generally, a 1% solution will produce a film about 5  $\mu\text{m}$  thick, and a 6% solution yields a film thickness of about 50  $\mu\text{m}$ . However, when the object on which the fluoropolymer is deposited is fibrous or porous, the fluoropolymer penetrates into the object. Commonly owned and copending PCT International Application No. PCT/US98/26903 and U.S. Patent Application Serial No. 215,441 describe the penetration of fluoropolymer solutions into pores in thermal spray coatings which are used for corrosion protection. Fluorine x-ray fluorescence micrographs were used to demonstrate fluoropolymer penetration into the pores.

Useful herein are solutions of fluoropolymers with molecular weights in the range of from 200,000 to 400,000. These fluoropolymers are known to have excellent chemical resistance; and their solutions generally have relatively low viscosities, on the order of about 0.060-0.300 pascal seconds (60 to 300 centipoise) at shear rates from about 50 to 300  $\text{sec}^{-1}$ , which enables them to flow into the pores. The location of the fluoropolymer in the pores is also important because, unlike purely surface films, the material is not easily abraded or worn away. Rather, the fluoropolymer in the pore is protected from abrasion by the surrounding porous coating as well as any surface coating. Moreover, the fibers of the substrate are interconnected with fluoropolymer, thereby strengthening the article.

Treating paper with fluoropolymer solutions can significantly increase the tensile strength of paper, as shown in Example 2 below, as well as typically impart other desirable properties. The films or deposits have very low surface energies compared to untreated paper (e.g., in the range of 15 to 19 dynes/cm). Thus, they are generally difficult to wet with liquids such as water. Solid deposits like dirt, dust or inks will not easily adhere to such surfaces. The permeation of aggressive chemical constituents of the environment which may degrade or corrode the object is retarded, as the solutions penetrate into the bulk of the material as well as depositing on the surface. Finally, the fluoropolymer film or deposit itself is very inert to degradation from environmental chemicals.

One use for this invention is the preservation of a variety of papers, including books, newspaper pages and documents. As paper ages, it frequently becomes more brittle. This is a problem for historic books and documents of great age. Treatment of these papers with the fluoropolymer solutions, as described in this invention, have been shown to increase the tensile strength of the paper, as shown in the Examples below. As outlined in Examples 1 and 2 below, a paper or book can be dipped into the fluoropolymer/perfluoroalkane solution and air dried

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to remove the solvent. The treated papers do not absorb water and therefore do not fall apart when immersed in water. No radical change in appearance or feel of the paper is normally noted for those with relatively low gloss, such as newsprint or copier paper. For glossier papers, a thin external film of polymer can ordinarily be seen on close visual inspection. Typically, no blurring or removal of ink is noted. Writing on treated paper with soft pointed, felt tip pens is typically more difficult than on untreated paper, although it is still relatively easy to mark with hard pointed, ball point pens. In general, it is more difficult to get treated articles dirty, and easier to clean them.

The treatment of the paper is considered reversible. The deposited polymer can be substantially removed by treating the treated paper with pure solvent to redissolve the fluoropolymer.

Textiles are fibrous materials, somewhat similar to paper in their behavior when exposed to these fluoropolymer solutions. These solutions penetrate into the weave, and upon drying leave fluoropolymer deposits, thereby increasing the strength of the textile. The textile material is made resistant to water or liquid absorption. Dyes used on the textile will not be affected by the solvent used, and the textile will be resistant to staining. A fluoropolymer film formed around the fibers protects them from environmental degradation by limiting the permeation of environmental chemicals and gasses through the film. The process of this invention would be useful in preserving historical and heirloom textiles and garments, such as tapestries, costumes, wedding gowns and the like.

Paintings consist of paint or ink on a porous or fibrous substrate like canvas or paper. The fluoropolymer solution will deposit a film around the paint or ink which protects it from environmental degradation. The porous or fibrous substrate will be come infiltrated with the polymer. The strength of the material would be increased. The painting would be resistant to water or liquid absorption, as well as staining.

The treatment of this invention is particularly useful for paper or textile having images made of ink, pigment or dye which is soluble in water, but not in perfluoroalkane solvents.

A variety of amorphous fluoropolymers may be used as the strengthener of this invention. This includes fluorine-containing homopolymers and co-polymers which are soluble at 0.5% by weight or greater in the solvent. An amorphous fluoropolymer is one which does not contain significant amounts of crystallinity when measured by DSC, or whose heat of melting is less than 2 J/g.

Suitable fluoropolymers include amorphous fluoropolymers which are copolymers containing units from functional fluorinated comonomers or

nonfunctional comonomers. Examples are copolymers of tetrafluoroethylene (TFE) with functional or non-functional monomers such as fluoroolefins having from 2 to 8 carbon atoms and fluorinated alkyl vinyl ether in which the alkyl group contains from 1 to 5 carbon atoms (e.g., 1, 3, 4 or 5 carbon atoms).

- 5 Examples of the non-functional monomers include hexafluoropropylene (HFP), chlorotrifluoro ethylene (CTFE), perfluoro(ethyl vinyl ether) (PEVE), perfluoro(methyl vinyl ether) (PMVE) and perfluoro(propylene vinyl ether) (PPVE). Functional monomers include, for example, perfluoroethyl vinyl ether (EVE),  $\text{CF}_2=\text{CFOCF}_2\text{CF}(\text{CF}_3)\text{OCF}_2\text{CF}_2\text{COOCH}_3$ ,  
10  $\text{CF}_2=\text{CFOCF}_2\text{CF}(\text{CF}_3)\text{OCF}_2\text{CF}_2\text{SO}_2\text{F}$ ,  $\text{CF}_2=\text{CFOCF}_2\text{CF}(\text{CF}_3)\text{OCF}_2\text{CF}_2\text{CN}$ ,  $\text{C}_3\text{N}_3(\text{CF}_2\text{CF}_2\text{OCF}(\text{CF}_3)\text{CF}_2\text{OCF}=\text{CF}_2)_3$  (EVE-triazine),  $\text{CF}_2=\text{CFOCF}_2\text{CF}(\text{CF}_3)\text{OCF}_2\text{CF}_2\text{CH}_2\text{OH}$ ,  $\text{CF}_2=\text{CFOCF}_2\text{CF}(\text{CF}_3)\text{OCF}_2\text{CF}_2\text{CH}_2\text{PO}_2(\text{OH})_2$  (EVE-P),  $\text{CF}_2=\text{FOCF}_2\text{CF}(\text{CF}_3)\text{OCF}_2\text{CF}_2\text{COOH}$ , and 4,5-difluoro-2,2-bis(trifluoromethyl)-  
15 1,3-dioxole (PDD). Commercially available materials include those from DuPont, Wilmington, DE: Teflon® SF60 (TFE/PMVE/PEVE, DuPont, Wilmington DE), Teflon® SF61 (TFE/PMVE/PEVE/EVE-P), Teflon® SF50 (TFE/HFP), Teflon® AF1600, 1601 and 2400 (PDD/TFE), and Teflon® AF2130 (PDD/CTFE); and those from Asahi Glass, Japan: Cytop®.

- 20 Of note are amorphous fluoropolymers which comprise copolymerized units of TFE and PEVE. Amorphous copolymers of tetrafluoroethylene and perfluoro(ethyl vinyl ether) are disclosed in U.S. Patent Nos. 5,478,905, 5,637,663 and 5,663,255, and in commonly owned, co-pending U.S. Patent Application Serial No. 08/929,213, which are incorporated herein by reference. Copolymers  
25 including units from TFE and PEVE may also include units from one or more additional fluorinated monomers. A preferred additional monomer is PMVE. When perfluoro(methyl vinyl ether) is present in the fluoropolymer, perfluoro(ethyl vinyl ether) is preferably at least 15% of the combined weight of the combined perfluoro(ethyl vinyl ether) and perfluoro(methyl vinyl ether).

- 30 Also of note are amorphous fluoropolymers which comprise copolymerized units of TFE and PDD, or CTFE and PDD. Examples of these copolymers are known collectively as Teflon® AF, available from DuPont Company, Wilmington, DE. Various grades are available, including Teflon® AF1600, 1601 and 2400 (PDD/TFE) and Teflon® AF2130 (PDD/CTFE).

- 35 A perfluoroalkane solvent is a non-aqueous solvent in which a perfluoroalkane is the primary component. Suitable solvents include perfluorinated alkanes such as perfluorooctane. Suitable solvents also include mixtures which include perfluoroalkanes, such as FC-75 and FC-40 (3M,

Minneapolis, MN). In general, the perfluoroalkane solvents used in these solutions are not considered aggressive to many paper and textile articles. Most inks will not dissolve in perfluorinated solvents. Similarly, many substrates are unaffected by exposure to these solvents.

The fluoropolymer solutions may be applied to the fibrous articles by common coating methods, including but not limited to spray application, dipping and brushing. After application of the solutions, the articles can be dried by conventional methods (e.g., air or vacuum drying).

### EXAMPLES

#### Fluoropolymer Solution Preparation:

Teflon® AF solutions were used in the examples below, and were used as received from E. I. du Pont de Nemours and Company, Wilmington, DE, unless otherwise noted. To dilute the Teflon® AF2130, solvent (FC-75, 3M, Minneapolis, MN) was weighed and was placed into a container, with the calculated amount of Teflon® AF2130 added to the solvent. The samples were mixed before use.

#### EXAMPLE 1

##### Paper Treatment

Several types of paper, including newsprint and copier paper were dipped for about 30 seconds into several Teflon® AF solutions, having concentrations between 1 and 6% solids by weight, such that about half the sheet was impregnated with the solution. The papers were removed from the solution and dried for about 30 minutes. The entire sheets of paper were immersed in water at room temperature. The treated portion emerged in undamaged condition while the untreated paper fell apart. No dissolution or blurring of ink was observed in the treated portion of the newsprint.

#### EXAMPLE 2

##### Strength Testing of Paper

The strength of treated paper was compared to that of an untreated paper. Strips of paper (20 pound White Wove, Gilbert, Inc., Menasha, WI) about 2.5 cm (1 inch) wide, 20.3 cm (8 inches) long, and 0.010 cm (0.004 inches) thick, were immersed for 10 minutes in solutions with varying concentrations of fluoropolymer. The strips were removed and dried, leaving a fluoropolymer deposit in the paper. The tensile strengths of the strips were measured using a model 1122 Instron test machine (Instron Corp., Canton, MA) and ASTM Method D 828 procedures (the test bars were held in grips with a separation of 5 inches, and the cross head speed was 2 inches/minute). The fluoropolymer treatment



increased the strength of the paper. The results are shown in Table 1 below, as well as in Figure 1.

TABLE 1

| Fluoropolymer<br>Solution Identification | Concentration of<br>Fluoropolymer, % | MPa  | Strength,<br>Ksi | % Increase in<br>Strength vs. Control |
|------------------------------------------|--------------------------------------|------|------------------|---------------------------------------|
| None                                     | 0                                    | 44.0 | 6.38             | ---                                   |
| TEFLON® AF1600                           | 3                                    | 51.2 | 7.42             | 16.3                                  |
| TEFLON® AF1601                           | 6                                    | 56.1 | 8.14             | 27.6                                  |
| TEFLON® AF2400                           | 1                                    | 48.9 | 7.09             | 11.1                                  |
| TEFLON® AF2130                           | 6                                    | 59.3 | 8.60             | 34.8                                  |
| TEFLON® AF2130                           | 3                                    | 59.0 | 8.55             | 34.0                                  |
| TEFLON® AF2130                           | 1                                    | 47.8 | 6.94             | 8.8                                   |

### CLAIMS

What is claimed is:

1. A method for strengthening a cellulosic paper or textile article, comprising the steps of:
  - (a) applying to the article a solution of an amorphous fluoropolymer in a perfluoroalkane solvent; and
  - (b) drying the article so that the solvent is essentially removed.
2. The method of Claim 1 wherein the fluoropolymer has a molecular weight in the range of from 200,000 to 400,000.
3. The method of Claim 1 wherein the fluoropolymer is a copolymer of 4,5-difluoro-2,2-bis(trifluoromethyl)-1,3-dioxole with either tetrafluoroethylene or chlorotrifluoroethylene.
4. The method of Claim 1 wherein the fluoropolymer is a copolymer of tetrafluoroethylene with a monomer selected from fluoroolefins having from 2 to 8 carbon atoms and fluorinated alkyl vinyl ethers where the alkyl group contains from 1 to 5 carbons.
5. A strengthened cellulosic paper or textile article, comprising:
  - (i) a fibrous cellulosic paper or textile substrate; and
  - (ii) amorphous fluoropolymer interconnecting fibers of said substrate.
6. The strengthened cellulosic paper or textile article of Claim 5 wherein the fluoropolymer has a molecular weight in the range of from 200,000 to 400,000.
7. The strengthened cellulosic paper or textile article of Claim 5 wherein the fluoropolymer is a copolymer of 4,5-difluoro-2,2-bis(trifluoromethyl)-1,3-dioxole with either tetrafluoroethylene or chlorotrifluoroethylene.
8. The strengthened cellulosic paper or textile article of Claim 5 wherein the fluoropolymer is a copolymer of tetrafluoroethylene with a monomer selected from fluoroolefins having from 2 to 8 carbon atoms and fluorinated alkyl vinyl ethers where the alkyl group contains from 1 to 5 carbons.
9. A strengthened article of paper or textile, comprising:
  - a) a pattern or printing, and
  - b) an amorphous fluoropolymer coating applied from an amorphous fluoropolymer solution such that the information conveyed by the pattern or printing is not obscured or distorted.
10. The process of treating a patterned or printed article of paper or textile comprising: applying to the article a solution of an amorphous fluoropolymer in a perfluoroalkane solvent; and drying the article so that the solvent is essentially removed.

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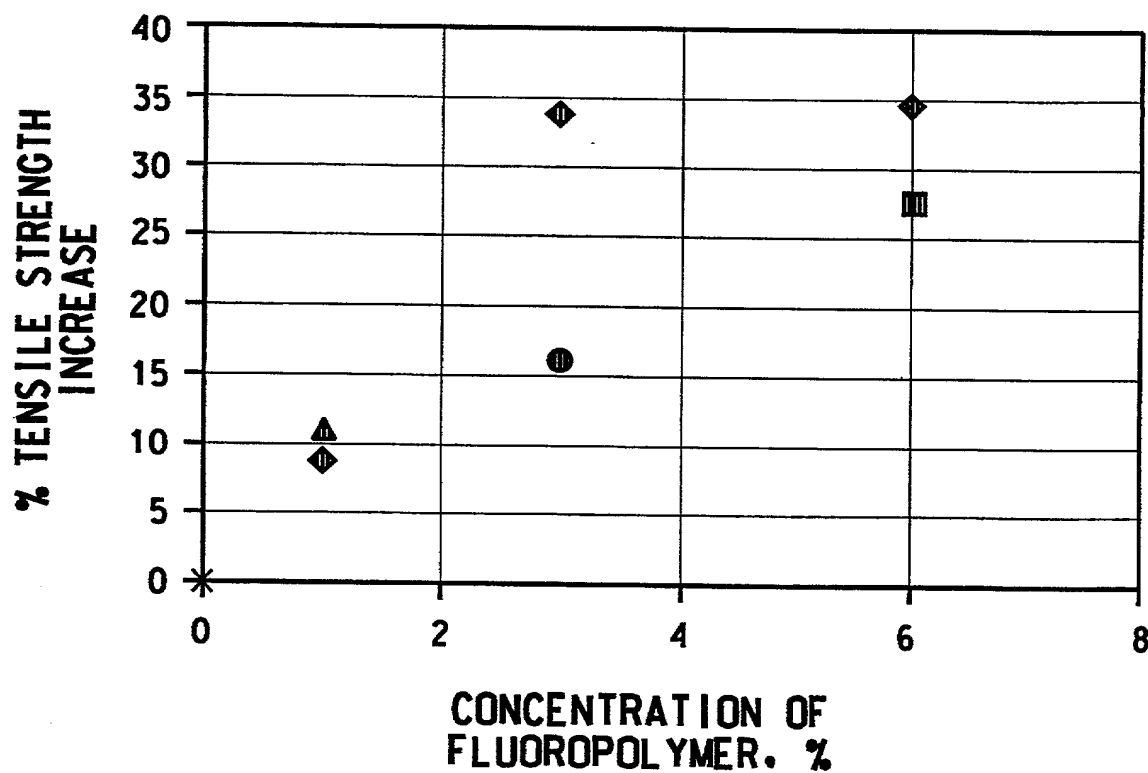


FIG. 1

\* NONE

● TEFLON<sup>®</sup> AF1600■ TEFLON<sup>®</sup> AF1601▲ TEFLON<sup>®</sup> AF2400◆ TEFLON<sup>®</sup> AF2130

**DECLARATION and POWER OF ATTORNEY**

As a below-named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**Preservation of Paper and Textile Materials**

the specification of which is attached hereto unless the following box is checked:

☒ was filed on **13 JUNE 2000** as U.S. Application No. \_\_\_\_\_ or PCT International Application No. **PCT/US00/16234** and was amended on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is known to me to be material to patentability as defined in 37 CFR § 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

| Application No. | Country | Filing Date | Priority Claimed (Yes/No) |
|-----------------|---------|-------------|---------------------------|
|-----------------|---------|-------------|---------------------------|

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States Provisional Application(s) listed below.

| U.S. Provisional Application No. | U.S. Filing Date |
|----------------------------------|------------------|
| 60/139,651                       | 17 JUNE 1999     |

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s), or § 365(c) of any PCT International Application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application or PCT International Application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is known to me to be material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

| Application No. | Filing Date | Status (patented, pending or abandoned) |
|-----------------|-------------|-----------------------------------------|
|-----------------|-------------|-----------------------------------------|

**POWER OF ATTORNEY:** I hereby appoint the following attorney(s) and/or agent(s) the power to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:Name: **BARBARA C. SIEGELL**Registration No.: **30,684**

Send correspondence and direct telephone calls to:

**BARBARA C. SIEGELL****E. I. du Pont de Nemours and Company  
Legal - Patents  
Wilmington, DE 19898, U.S.A.**Tel. No.  
**(302) 992-4931**Fax No.  
**(302) 892-7949**

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

**INVENTOR(S)**

|                         |                                                                        |                                             |                                                              |
|-------------------------|------------------------------------------------------------------------|---------------------------------------------|--------------------------------------------------------------|
| Full Name of Inventor   | Last Name<br><b>BIRCHENALL</b>                                         | First Name<br><b>ANDREW</b>                 | Middle Name<br><b>KELSEY</b>                                 |
|                         | Signature (please sign full name):<br><i>Andrew Kelsey Birchennall</i> |                                             | Date:<br><b>7/28/00</b>                                      |
| Residence & Citizenship | City<br><b>MIDDLETOWN</b>                                              | State or Foreign Country<br><b>DELAWARE</b> | Country of Citizenship<br><b>U.S.A.</b> <b>DE</b>            |
| Post Office Address     | Post Office Address<br><b>1350 CEDAR LANE ROAD</b>                     | City<br><b>MIDDLETOWN</b>                   | State or Country<br><b>DELAWARE</b> Zip Code<br><b>19709</b> |

☐ Additional Inventors are being named on separately numbered sheets attached hereto.

GENERAL POWER OF ATTORNEY  
(Concerning Several International Patent Applications)

The undersigned, Vernon R. Rice, Vice President and Assistant General Counsel of E. I. DU PONT DE NEMOURS AND COMPANY, 1007 Market Street, Wilmington, Delaware 19898 USA ("DuPont"), hereby confirms that the power to sign for DuPont has been granted to various individuals (as set forth in the attached excerpt from DuPont's Patent Board Rules of Procedure (January 1988), Appendix Section III.A.4), including the Chairman, Vice-Chairman, and those individuals who are Assistant Secretaries of the Patent Board. Currently these Assistant Secretaries are:

Roger A. Bowman  
Linda J. Davis  
John E. Griffiths

Barbara J. Massie  
Miriam D. Meconnahey  
Deborah A. Meginniss

In addition, the authority to act on behalf of DuPont before the competent International Authorities in connection with any and all international patent applications filed by it with the United States as Receiving Office and to make or receive payments on its behalf is hereby granted to:

|                        |        |                          |        |
|------------------------|--------|--------------------------|--------|
| Beardell, Lori Y.      | 34,293 | Katz, Elliott A.         | 26,396 |
| Belopolsky, Inna       | 43,319 | Kelly, Patricia L.       | 39,247 |
| Benjamin, Steven C.    | 36,087 | King, Karen K.           | 34,850 |
| Birch, Linda D.        | 38,719 | Kuller, Mark D.          | 31,925 |
| Bowen, Jr., Alanson G. | 24,027 | Krukiel, Charles E.      | 27,344 |
| Christenbury, Lynne M. | 30,971 | Jarnholm, Arne R.        | 30,396 |
| Cotreau, William J.    | 36,490 | Langworthy, John A.      | 32,255 |
| Deitch, Gerald E.      | 30,457 | Lerman, Bart E.          | 31,897 |
| Deshmukh, Sudhir       | 33,677 | Levitt, Cary A.          | 31,848 |
| Dobson, Kevin S.       | 40,296 | Magee, Thomas H.         | 27,355 |
| Duffy, Roseanne R.     | 33,869 | Mayer, Nancy S.          | 29,190 |
| Edwards, Mark A.       | 39,542 | Medwick, George M.       | 27,456 |
| Estrin, Barry          | 26,452 | Morrissey, Bruce W.      | 30,663 |
| Evans, Craig H.        | 31,825 | Reynolds, Stephen E.     | 37,580 |
| Fair, Tamera L.        | 35,867 | Rizzo, Thomas M.         | 41,272 |
| Feltham, S. Neil       | 36,506 | Santopietro, Lois A.     | 36,264 |
| Floyd, Linda Axamethy  | 33,692 | Schaeffer, Andrew L.     | 33,605 |
| Fricke, Hilmar L.      | 22,384 | Sebree, Chyrrea J.       | 45,348 |
| Furr, Robert B.        | 32,985 | Shay, Lucas K.           | 34,724 |
| Golian, Andrew G.      | 25,293 | Shipley, James E.        | 32,003 |
| Golian, Paul D.        | 42,591 | Siegell, Barbara C.      | 30,684 |
| Gorman, Thomas W.      | 31,959 | Sinnott, Jessica M.      | 34,015 |
| Gould, David J.        | 25,338 | Steinberg, Michael A.    | 43,160 |
| Griffiths, John E.     | 32,647 | Steinberg, Thomas W.     | 37,013 |
| Hamby, Jane O.         | 32,872 | Stevenson, Robert B.     | 26,039 |
| Hamby, William H.      | 31,521 | Strickland, Frederick D. | 39,041 |
| Heiser, David E.       | 31,366 | Tulloch, Rebecca W.      | 36,297 |
| Hendrickson, John S.   | 30,847 | Walker, P. Michael       | 32,602 |
| Joung, J. Kenneth      | 41,881 | Wang, Chen               | 38,650 |

The undersigned ratifies fully all actions already taken by the above-named individuals in accordance with the authority granted hereby.

E. I. DU PONT DE NEMOURS AND COMPANY

By: 

Vernon R. Rice  
Vice President and Assistant General Counsel

0-9-01